[CONTRIBUTION FROM THE IPATIEFF HIGH PRESSURE AND CATALYTIC LABORATORY, DEPARTMENT OF CHEMISTRY, NORTHWESTERN UNIVERSITY]

Potassium-Catalyzed Reactions of α-Methylstyrene with Alkylbenzenes[1,2]

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α-Methylstyrene reacts readily with *n*-alkylbenzenes at 100–105° in the presence of dispersed potassium to form diphenylalkanes in high yield and purity. The reactions of α-methylsytrene with toluene, ethylbenzene, and *n*-propylbenzene result in the formation of 1,3-diphenylbutane in 73%, 2,4-diphenylpentane in 85%, and 2,4-diphenylhexane in 75% yield, respectively. Some quantities of 2,5-diphenylhexane, 2-methyl-2,4-diphenylpentane, and 1-methyl-1,3-diphenylcyclopentane, derived from the interaction of two α-methylstyrene molecules, appear as by-products in all cases. By raising the reaction temperature to 125–130° and/or extending the reaction time, the products become somewhat more complex as a result of splitting and resynthesis reactions of the primary products.

In contrast to its behavior towards *n*-alkylbenzenes, α-methylstyrene does not undergo reaction with cumene in the absence of promoters. In the presence, however, of *o*-chlorotoluene a small yield of the monoadduct, 2-methyl-2,4-diphenylpentane, was obtained. The major reaction is the self condensation of α-methylstyrene to form 1-methyl-1,3-diphenylcyclopentane.

In previous papers of these series we reported that styrene[4] and *o*-, *m*- and *p*-methylstyrene[1] undergo reaction with alkylbenzenes to form addition compounds. This reaction, however, was accompanied by an extensive polymerization of the styrenes. It was also reported[5] that α-methylstyrene in the presence of sodium undergoes dimerization forming as the principal product 1-methyl-1,3-diphenylcyclopentane. As an extension of this study we are reporting the reaction of α-methylstyrene with alkylbenzenes in the presence of sodium and of potassium as catalysts.

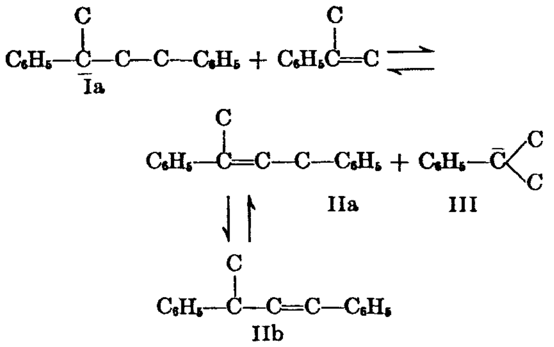
A preliminary experiment had shown that in the presence of sodium-benzylsodium catalyst[1,6] at 110° and three hours of contact time, only 5% of the α-methylstyrene reacted with toluene. The time needed for completion of this reaction was twenty hours. In the presence, however, of dispersed potassium and in the absence of the promoter the reaction proceeded quantitatively within two hours. For that reason the present study was carried out using potassium as a catalyst for the aralkylation reaction.

The experimental technique was similar to that described in the preceeding paper.[1] The reaction product was separated and analyzed by a combination of fractional distillation, selective hydrogenation, gas-liquid partition chromatography, and infrared and ultraviolet spectroscopy. The experimental results are summarized in Table I.

RESULTS

a. *Toluene and α-methylstyrene.* The main product of the reaction is 1,3-diphenylbutane (I), which was formed through the addition of a benzyl carbanion to the α-methylstyrene followed by protonation, according to the mechanism described previously.[1,7]

The intermediate carbanion formed in this addition (Ia) undergoes to a limited extent a hydride elimination reaction as indicated by the presence both of cumene and small amounts of olefins skeletally identical with 1,3-diphenylbutane:



(1)

The occurrence of hydride transfer reactions is evidenced also by the presence of 2-methyl-2,4-diphenylpentane (IV), which is probably formed by the addition of III to α-methylstyrene.

The main competing monoaddition reaction results from the dimerization of α-methylstyrene:

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(1) Paper XXI of the series "Base-catalyzed Reactions." For paper XX, see H. Pines and J. Shabtai, *J. Org. Chem.*, 26, 4220 (1961).

(2) This research was supported by a grant from the Petroleum Research Fund administered by the American Chemical Society. Grateful acknowledgment is hereby made to the donors of the fund.

(3) On leave of absence from the Weizmann Institute of Science, Rehovoth, Israel.

(4) H. Pines and D. Wunderlich, *J. Am. Chem. Soc.*, **80**, 6001 (1958).

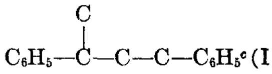
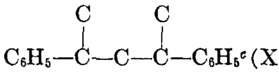
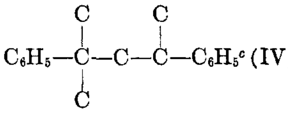
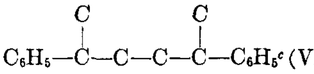
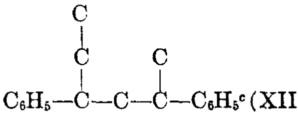
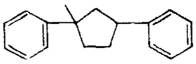
(5) M. Kolobielski and H. Pines, *J. Am. Chem. Soc.*, **79**, 5820 (1957).

(6) H. Pines and H. E. Eschinazi, *J. Am. Chem. Soc.*, **77**, 6314 (1955).

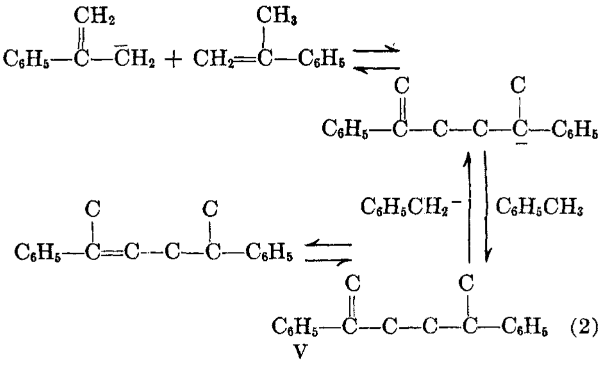
(7) For references to other papers see H. Pines and L. A. Schaap, "Base Catalyzed Reactions of Hydrocarbons" in *Advances in Catalysis*, Vol. XII, Academic Press, New York, 1960.

TABLE I

COMPOSITION OF PRODUCTS OBTAINED FROM THE POTASSIUM-CATALYZED REACTIONS OF α-METHYLSTYRENE WITH ALKYLBENZENES

Exp. No.	1	2	3	4	5	6	7
C_6H_5R used,[a] R =	CH_3	CH_3	C_2H_5	C_2H_5	n-C_3H_7	n-C_3H_7	i-C_3H_7
Temperature,	105	105	105	125	105	125	125
Reaction time, hr.	2	5	2	2	2	5	4
Component, wt. %							
Toluene	[b]	[b]	0.2	0.4		Trace	4.1
Ethylbenzene	0.3	2.5	[b]	[b]	Trace	1.3	3.7
Cumene	1.1	1.2	3.9	4.0	4.5	5.8	[b]
sec-Butylbenzene						0.3	
$C_6H_5—C—C—C—C_6H_5{}^{c}$ (X)		4.7		0.3			1.8
 $C_6H_5—\overset{C}{\overset{\vert}{C}}—C—C—C_6H_5{}^{c}$ (I)	72.8	65.0	1.2	6.7		0.2	2.5
 $C_6H_5—\overset{C}{\overset{\vert}{C}}—C—\overset{C}{\overset{\vert}{C}}—C_6H_5{}^{c}$ (XI)			84.5	75.3	0.4	14.5	6.7
 $C_6H_5—\underset{\underset{C}{\vert}}{\overset{C}{\overset{\vert}{C}}}—C—\overset{C}{\overset{\vert}{C}}—C_6H_5{}^{c}$ (IV)	0.5	0.5	Trace	Trace			8.8
 $C_6H_5—\overset{C}{\overset{\vert}{C}}—C—C—\overset{C}{\overset{\vert}{C}}—C_6H_5{}^{c}$ (VI)	3.7	3.4	2.9	2.6	9.0	18.0	15.3
 $C_6H_5—\overset{C}{\overset{\vert}{\overset{C}{\overset{\vert}{C}}}}—C—\overset{C}{\overset{\vert}{C}}—C_6H_5{}^{c}$ (XIII)					74.6	40.7	
 (XII)	0.3	0.6	4.5	7.8	6.5	11.7	53.0
Diadduct 1[d]	8.8	8.4					
Diadduct 2[e]	9.8	9.5					
High-boiling residue	2.7	4.2	2.8	2.9	5.0	7.5	4.1

[a] In each experiment were used: 24 g. (0.2*M*) of α-methylstyrene, 1.2*M* of alkylbenzene and 2 g. of potassium. [b] No attempt was made to determine the amount of this hydrocarbon formed during the reaction because of its use as a reagent. [c] Skeletally identical olefins are included in the yield; see experimental part. [d] Assigned structure: 1,3-diphenyl-2-benzylbutane (VII). [e] Assigned structure: 2,4,6-triphenylheptane (VIII).



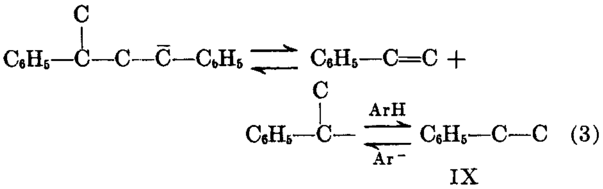
The intermediate carbanion may undergo cyclization to form 1-methyl-1,3-diphenylcyclopentane.[5] The open-chain dimer V can undergo to some extent hydrogenation to 2,5-diphenylhexane (VI), by accepting a hydride ion, followed by proton abstraction.

The formation of 1,3-diphenyl-2-benzylbutane (VII) can be attributed to the reaction of toluene with compounds IIa and IIb.

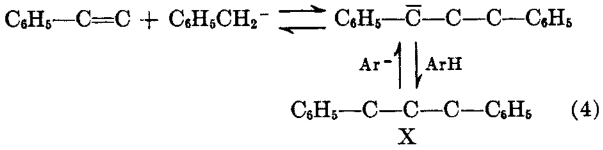
Diadduct 2 [2,4,6-triphenylheptane (VIII)] is most likely formed by metalation of the main product, 1,3-diphenylbutane ($C_6H_5—\overset{C}{\overset{\vert}{C}}—C—\underline{C}—C_6H_5$), followed by interaction with a α-methylstyrene molecule.

The second possibility of a similar reaction initiated by the formation of a tertiary benzylic carbanion ($C_6H_5\overset{C}{\overset{\vert}{\underline{C}}}—C—C—C_6H_5$) is excluded, because of (a) the low rate of metalation of the tertiary carbon in 1,3-diphenylbutane and (b) a still lower rate of addition of such a tertiary carbanion to α-methylstyrene (see below, reactions of cumene with α-methylstyrene).

By increasing the reaction time from two to five hours (Exp. 2), that is, by continuing the reaction after the complete conversion of the α-methylstyrene, the product becomes somewhat more complex. The decrease in the amount of 1,3-diphenylbutane and the simultaneous appearance of 1,3-diphenylpropane and ethylbenzene in the product point to the occurrence at this stage of carbanion splitting as well as resynthesis reactions:



$$C_6H_5-\overset{C}{C}-C-\bar{C}-C_6H_5 \rightleftharpoons C_6H_5-C=C + C_6H_5-\overset{C}{C}- \underset{Ar^-}{\overset{ArH}{\rightleftharpoons}} \underset{IX}{C_6H_5-C-C} \quad (3)$$



$$C_6H_5-C=C + C_6H_5CH_2^- \rightleftharpoons C_6H_5-\bar{C}-C-C-C_6H_5 \; \underset{Ar^-}{\uparrow}\,\underset{ArH}{\downarrow} \; \underset{X}{C_6H_5-C-C-C-C_6H_5} \quad (4)$$

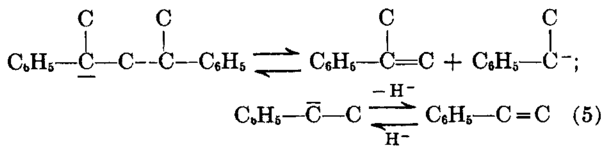
ArH = an alkylaromatic.

b. *Ethylbenzene and α-methylstyrene.* The expected monoadduct, 2,4-diphenylbutane (XI), was formed in 84.5% yield.

The high yield of the monoadduct in the absence of any diadduct could be attributed to the difficulty of the addition of the intermediate tertiary carbanion $(C_6H_5\overset{C}{C}-C-\overset{C}{C}-C_6H_5)$ to α-methylstyrene, as it will be shown in the experiments where cumene was used as an aralkylating agent.

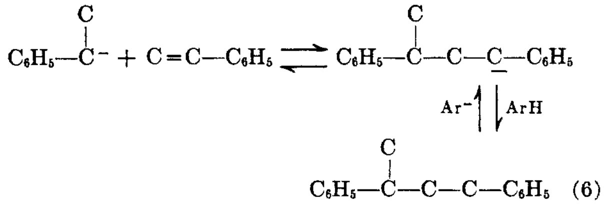
As in the previous case the main competing reaction was the dimerization of α-methylstyrene to form both open chain and cyclic dimers. The cyclization reaction, resulting in the formation of 1-methyl-1,3-diphenylcyclopentane (XII), involved the intermediate carbanion formed in reaction (2) and occurred according to the mechanism described previously.[5]

The effect of temperature upon the occurrence of secondary reaction had been studied. By raising the temperature from 105° to 125° (Exp. 4) the yield of side reaction increases considerably at the expense of the monoadduct (XI). The increase in the formation of toluene, 1,3-diphenylpropane, and 1,3-diphenylbutane can be explained by scission of the monoadduct, followed by a recombination of the fragments:



$$C_6H_5-\underset{-}{\overset{C}{C}}-C-\overset{C}{C}-C_6H_5 \rightleftharpoons C_6H_5-\overset{C}{C}=C + C_6H_5-\overset{C}{C}^-;$$

$$C_6H_5-\bar{C}-C \underset{H^-}{\overset{-H^-}{\rightleftharpoons}} C_6H_5-C=C \quad (5)$$



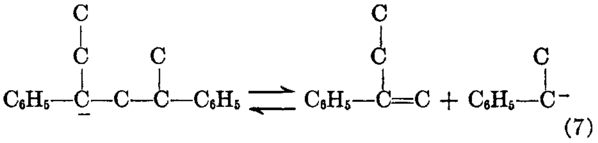
$$C_6H_5-\overset{C}{C}^- + C=C-C_6H_5 \rightleftharpoons C_6H_5-\overset{C}{C}-C-\underset{-}{C}-C_6H_5 \; \underset{Ar^-}{\uparrow}\,\underset{ArH}{\downarrow} \; C_6H_5-\overset{C}{C}-C-C-C_6H_5 \quad (6)$$

The 1,3-diphenylbutane thus formed can give by splitting some toluene, which then interacts with styrene to form 1,3-diphenylpropane according to reaction (4).

c. *n-Propylbenzene and α-methylstyrene.* Two experiments were performed with α-methylstyrene in the presence of *n*-propylbenzene. In the first experiment (Exp. 5) the product consisted mainly of the expected normal addition product, 2,4-diphenylhexane (74.6 wt. %). The extent of the competing dimerization reactions leading to the formation of V and XII is however considerably higher than that observed in the corresponding reaction (Exp. 3) in the presence of ethylbenzene. This would seem to indicate that the rate of addition of *n*-propylbenzene to α-methylstyrene is somewhat lower than that of ethylbenzene.

As in the case of ethylbenzene no formation of diadducts was observed to any appreciable extent.

In Experiment 6 both the temperature and reaction time were increased (125°, five hours). As expected, this double change in conditions resulted in a higher extent of splitting reactions, the main secondary compound produced being 2,4-diphenylpentane.



$$C_6H_5-\underset{-}{\overset{\overset{C}{C}}{C}}-C-\overset{C}{C}-C_6H_5 \rightleftharpoons C_6H_5-\overset{\overset{C}{C}}{C}=C + C_6H_5-\overset{C}{C}^- \quad (7)$$

The carbanion thus formed can interact with α-methylstyrene to form the above-mentioned compound. The α-methylstyrene can be supplied in the late stage of the reaction by the alternative reversible splitting of the primary monoadduct.

The occurrence of reaction (7) is supported by the appearance both of ethylbenzene and a small amount of *sec*-butylbenzene in the product. The main part of the α-ethylstyrene formed probably reacts to form higher boiling products.

A second feature of the reaction at the higher temperature is the considerable increase in the amount of the open-chain and cyclic dimers of α-methylstyrene (about 30% by weight of the total product).

d. *Cumene and α-methylstyrene.* In contrast to its behavior towards toluene, ethylbenzene, and *n*-propylbenzene, α-methylstyrene does not interact with cumene in the absence of promoters. On the other hand, in the presence of cumene and of *o*-chlorotoluene as a promoter, α-methylstyrene reacts to form mainly 1-methyl-1,3-diphenylcyclopentane by self condensation and cyclization. The total amount of this cyclic dimer and that of its open-chain precursor, 2,5-diphenylhexene (plus 2,5-diphenylhexane), accounts for nearly 70% by weight of the total product. The expected monoadduct, 2-methyl-2,4-diphenylpentane, amounted to only 8.8% of the total product. The cumyl carbanion required could be formed either from cumene or from α-methylstyrene by hydrogen transfer. The material balance of the experiment shows that about 80% of the compound has been derived from cumene. The limited extent of aralkylation of cumene, as compared with toluene, ethylbenzene, and *n*-propylbenzene, could be explained by a possible steric interference in the addition of the tertiary cumyl carbanion to α-methylstyrene.

Conclusions. The aralkylation of α-methylstyrene in the presence of potassium is not accompanied by an appreciable formation of high molecular weight polymers.

The yield of aralkylated α-methylstyrene is high in the case of toluene, ethyl-, and *n*-propylbenzene; it falls abruptly when isopropylbenzene is used. The competing reaction, the dimerization of α-methylstyrene, increases with the length and branching of the alkyl group of the aralkylating agent. In the case of cumene, the formation of 1-methyl-1,3-diphenylcyclopentane, a product of dimerization of α-methylstyrene, amounted to 53% of the product.

Toluene reacts with α-methylstyrene to form mono- and diadducts; diadducts are absent when higher alkylbenzenes are used. This is a further proof that a tertiary carbanion does not add readily to α-methylstyrene.

Secondary reactions take place when the contact time and/or the temperature of the reaction is increased. The formation of the secondary reaction products can be explained by carbanion scission of the monoadducts followed by a recombination of the fragments.

EXPERIMENTAL

Apparatus and procedure. The experiments were carried out in an apparatus similar to the one described previously.[1] Thirty grams of the purified alkylbenzene (previously washed with concentrated sulfuric acid to remove peroxides, distilled, and dried over sodium) was first introduced in the flask and 2 g. of freshly cut potassium added to it. The potassium was weighed and cut in every case under a protecting layer of the alkylbenzene. The air in the flask was displaced by a stream of helium, the mixture was heated to the desired temperature and afterwards stirred for 30 min. An additional 30 g. of the alkylbenzene was then added dropwise and the stirring was continued for another 30 min. The potassium was dispersed in this way in the form of a very fine white powder (in some cases the powder becomes slightly violet tinted). Twenty-four grams of freshly distilled α-methylstyrene, dissolved in the remaining part of the alkylbenzene, was then added in an interval of 45 to 60 min. The color of the mixture changed rapidly from white to dark brown or black during the addition. The mixture was then stirred at constant temperature for the desired period. The reaction product was separated according to the procedure described previously.[1]

TABLE II

RETENTION VOLUMES OF DIPHENYLALKANES RELATIVE TO *n*-HEXYLBENZENE[a]

Compound	$Rv/Rv_{n\text{-hexylbenzene}}$
1,3-Diphenylpropane	5.4
1,3-Diphenylbutane	5.8
2,4-Diphenylpentane	6.3
2,4-Diphenylhexane	7.2
2-Methyl-2,4-diphenylpentane	7.4
2,5-Diphenylhexane	8.4
1-Methyl-1,3-diphenylcyclopentane	14.5

[a] Temp. 215°; 12-ft. column filled with 15% silicon (Dow-Corning 550 fluid) on 30–60 mesh Celite; helium flow rate 125 ml./min.

The Analytical procedure was the same as that described in the previous paper of this series.[1] The relative retention volumes of the main diphenylalkanes studied are given in Table II.

Experiment 1. α-Methylstyrene-toluene. After completing the reaction, the excess of alkylbenzene was distilled at atmospheric pressure. The distillate, 94.5 g., was composed of: toluene, 99.4%; cumene, 0.5%; ethylbenzene, 0.1%. The solvent-free product, 37.4 g., was distilled fractionally, as follows:

No.	B.P.	n_D^{20}	Unsatn. (%)	Wt. %
1	113–114/1 mm.	1.5520	3.0	71.9
2a	115–130/1 mm.	1.5551	23.2	2.8
2b	130–145/1 mm.	1.5658	41.0	3.4
3	180–181/0.8 mm.	1.5733	6.4	9.1
4	205–206/0.8 mm.	1.5754	<0.1	10.0
Residue plus hold up		—	—	2.8

After hydrogenation, fraction 1 was identified as 1,3-diphenylbutane of 99.5% purity; b.p. 113°/1 mm., n^{20}D 1.5519 (reported,[4] b.p. 105°/1 mm., n_D^{20} 1.5520).

Anal. Calcd. for $C_{16}H_{18}$: C, 91.37; H, 8.63. Found: C, 91.33; H, 8.46.

The hydrogenated fractions 2a and 2b had the following composition, as determined by gas-liquid phase chromatography on a silicon column:

Component, Wt. %	Fraction 2a	Fraction 2b
1,3-Diphenylbutane	44.1	20.6
2-Methyl-2,4-diphenylpentane	11.4	6.7
2,5-Diphenylhexane	44.5	64.2
1-Methyl-1,3-diphenylcyclopentane	—	8.5

The main part of the 2,5-diphenylhexane in the examined hydrogenated fractions was derived from skeletally identical olefins, probably a mixture of 2,5-diphenyl-1-hexene and 2,5-diphenyl-2-hexene. The presence of components having a phenyl-conjugated double bond in the nonhydrogenated fractions 2a and 2b was shown by ultraviolet analysis. Fraction 2b, for example, showed λ_{max} 2490Å, ε 4840. A rough estimate of the olefin component in this fraction was made by using a compound of the same type (1-phenyl-3-*p*-tolylpropene, λ_{max} 2500 Å, ε 9000) as the reference. On this basis, the concentration of 2,5-diphenylhexene in fraction 2b was calculated to be about 50%. This accounts for nearly 80% of the 2,5-diphenylhexane found in the hydrogenated fraction. The estimation was in fairly good agreement with the extent of unsaturation of the sample (41%).

Fractions 3 and 4 were assumed to contain individual diadducts or mixtures of very close boiling isomers:

Anal. of fraction 3. Calcd. for $C_{23}H_{24}$: C, 91.94; H, 8.06; mol. wt., 300. Found: C, 91.83; H, 7.91; mol. wt., 296.

Anal. of fraction 4. Calcd. for $C_{25}H_{28}$: C, 91.40; H, 8.60; mol. wt., 328. Found: C, 91.50; H, 8.46; mol. wt., 334.

Examination of the infrared spectra of fractions 3 and 4 in the 1700–2000-cm.$^{-1}$ region shows in both cases the presence of a grouping of bands specific for compounds having monosubstituted benzene rings only.[8] Monosubstitution is evidenced also by the appearance of a strong absorption band at 693 cm.$^{-1}$ The data obtained for fractions 3 and 4 are in accord with structures assigned for the two possible types of diadduct—1,3-diphenyl-2-benzylbutane and 2,4,6-triphenylheptane, respectively.

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[8] C. W. Young, R. B. Du Vall, and N. Wright, *Anal. Chem.*, **23**, 709 (1951).

Experiment 4. α-Methylstyrene-ethylbenzene. The ethylbenzene fraction, 106 g., was recovered by distillation at 100 mm. This fraction was composed of: toluene, 0.15%; ethylbenzene, 98.35%; cumene, 1.5%. The higher boiling product, amounting to 40.6 g., was distilled and the following fractions collected:

No.	B.P.	n_D^{20}	Unsatn. (%)	Wt. %
1	118–119/1 mm.	1.5485	9.0	84.3
2	125–141/1 mm.	1.5662	8.8	12.7
Residue plus hold up		—	—	3.0

The hydrogenated fraction 1 was composed of: 2,4-diphenylpentane, 91.4%; 1,3-diphenylbutane, 8.2%; 1,3-diphenylpropane, 0.4%. In the corresponding fraction 1, in the experiment carried out at 105°, the purity of the 2,4-diphenylpentane after hydrogenation was about 99%; b.p. 117–118°/1 mm., n_D^{20}, 1.5485.

Anal. Calcd. for $C_{17}H_{20}$: C, 91.01, H, 8.99. Found: C, 91.01; H, 8.75.

Fraction 2 consisted of: 2,4-diphenylpentane, 15.8%; 2,5-diphenylhexane, 20.7%; 1-methyl-1,3-diphenylcyclopentane, 63.5%.

Experiment 5. α-Methylstyrene-n-propylbenzene. The recovered *n*-propylbenzene fraction, 128 g., had the following composition: *n*-propylbenzene, 98.7%; cumene, 1.2%; ethylbenzene, ~0.1%. The product., 39.5 g., was distilled and the following fractions were collected:

No.	B.P.	n_D^{20}	Unsatn. (%)	Wt. %
1	124–125/1 mm.	1.5457	3.5	85.3
2	128–140/1 mm.	1.5660	9.0	8.0
Residue plus hold up		—	—	6.7

The hydrogenated fractions had the following composition.

Component, Wt. %	Fraction 1	Fraction 2
2,4-Diphenylhexane	89.0	17.5
2,5-Diphenylhexane	8.5	24.2
1-Methyl-1,3-diphenylcyclopentane	2.5	58.3

The normal adduct, 2,4-diphenylhexane, which was obtained in pure form by precise distillation, had a b.p. of 124–125°/1 mm., n_D^{20}, 1.5460.

Anal. Calcd. for $C_{18}H_{22}$: C, 90.69; H, 9.31. Found: C, 90.56; H, 9.25.

Experiment 7. α-Methylstyrene-cumene. The recovered solvent (143.2 g.) had the following composition: toluene, 0.70%; ethylbenzene, 0.65%; cumene, 98.65%.

The solvent-free product, 25.4 g., was distilled fractionally as follows:

No.	B.P.	n_D^{20}	Unsatn. (%)	Wt. %
1	122–130/0.8 mm.	1.5578	22.7	45.0
2	140–145/0.8 mm.	1.5710	3.8	48.5
Residue plus holdup		—	—	6.5

The hydrogenated fractions had the following composition:

Component, Wt. %	Fraction 1	Fraction 2
1,3-Diphenylpropane	4.5	—
1,3-Diphenylbutane	6.2	—
2,4-Diphenylpentane	16.0	—
2-Methyl-2,4-diphenylpentane	19.0	2.2
2,5-Diphenylhexane	27.6	10.0
1-Methyl-1,3-diphenylcyclopentane	26.7	87.8

The relatively high degree of unsaturation of fraction 1 is due mainly to the presence of 2,5-diphenylhexene, the primary dimerization product of α-methylstyrene.

Source and synthesis of pure hydrocarbons. Pure samples (98% or more) of 1,3-diphenylpropane, 1,3-diphenylbutane, and 1-methyl-1,3-diphenylcyclopentane were available in this laboratory from previous work on the sodium-catalyzed reactions of styrene[4] and α-methylstyrene.[5] 2,5-Diphenylhexane and 2-methyl-2,4-diphenylpentane were prepared.

2,5-Diphenylhexane. A mixture of 2-phenylallyl bromide and 2-phenyl-1-bromopropene was prepared in 58% yield by the bromination of α-methylstyrene with *N*-bromosuccinimide according to the procedure described previously.[9] An ethereal solution of the bromides was then treated with magnesium in the presence of anhydrous cuprous chloride to form mainly 2,5-diphenyl-1,5-hexadiene and a smaller amount of the isomeric 2,5-diphenyl-2,4-hexadiene. The yield of the cut corresponding to 2,5-diphenyl-1,5-hexadiene was 51%; b.p. 145–147°/2 mm, m.p. 47–48° (reported,[9] b.p. 145–150°/2 mm., m.p. 47–48°). Six grams of the cut was dissolved in a mixture of 10 ml. of *n*-hexane and 10 ml. of ethanol and hydrogenated at 160° and 100 atm. pressure in the presence of a copper chromite catalyst. After removing the solvent, the 2,5-diphenylhexane was distilled *in vacuo*; b.p. 127–128°/0.8 mm., n_D^{20}, 1.5425.

Anal. Calcd. for $C_{18}H_{22}$: C, 90.69; H, 9.31. Found: C, 90.58; H, 9.20.

The purity of the compound, as determined by gas-liquid phase chromatography, was nearly 99%.

2-Methyl-2,4-diphenylpentane. A pure sample of 4-methyl-2,4-diphenyl-2-pentanol, 15 g., b.p. 146°/2 mm., n_D^{20} 1.5614 (available in this laboratory from previous work) was dissolved in 30 ml. of *tert*-butyl alcohol and dehydrated at 330° over activated alumina (Harshaw, $^1/_8$" tablets). The resulting olefin, b.p. 133–134°/1 mm., n_D^{20} 1.5730, was dissolved in a mixture of 10 ml. of *n*-hexane and 10 ml. of ethanol and hydrogenated at 160° at an initial pressure of 110 atm. using copper chromite as a catalyst. The 2-methyl-2,4-diphenylpentane distilled at 119°/0.8 mm., n_D^{20} 1.5487. The purity of the compound was about 99%.

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(9) H. Pines, H. Allul, and M. Kolobielski, *J. Org. Chem.*, 22, 1113 (1957).